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METHOD 548.1

I. SCOPE AND APPLICATION:

This method is for the identification and simultaneous measurement of endothall in drinking water sources and finished drinking water. This is a gas chromatographic/mass spectrometric (GCMS) method. However, a flame ionization detector (FID) may be utilized for the determination, but must be supported by an additional analysis using a confirmatory gas chromatographic column. The following compound can be determined using this method:

Analyte	Chemical Abstract Services Registry Numbers (CASRN)
Endothall	145-73-3

II. REAGENTS:

- Sodium Thiosulfate ($Na_2S_2O_3$) solution

III. MATERIALS:

- Amber glass borosilicate (250 ml or larger) sample bottle fitted with screw caps lined with Teflon. Collect duplicate bottles if bottles are less than 500 ml.
- Latex gloves
- Protective eyewear
- Plastic container for disposal of used pipette tips
- Disposable glass pipette and rubber bulb.
- Pool and Spa 3-Way Test Strips (Chem Lab Products, Inc.)
- Paper towels & Kim wipes
- Pliers

IV. PROCEDURE:

1. Remove any attachments such as hoses, screens or aeration devices on the faucet. Inspect the faucet for anything that may fall into the sample container.

2. Open the tap and allow the system to flush for about 10 minutes. This should be sufficiently long enough to allow the water temperature to stabilize and get a representative sample.

3. Adjust the water flow to about 1000 ml/minute or slow enough that no air bubbles purge the sample when collecting from the flowing stream.

4. Remove the cap from the sampling container. Do not rinse the container as it has already been acid rinsed and may already contain sodium thiosulfate as a preservative.

5. To fill, tip the bottle to about a 45° angle into the stream of water. Ensure the stream is sufficiently slow so as to be able to anticipate when the bottle is nearly full and thus avoid over flowing. Fill the bottle to within approximately ½ inch of the mouth.

6. Remove the bottle from the flow and recap. Invert the sample bottle five times.

7. Place a chlorine detector strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference chlorine range. A determination must be made within 30 seconds.

8. If no chlorine is detected, recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in the field notebook and place the sample bottle in the ice chest to cool to 4°C. Collect duplicate samples.

9. If chlorine is present, add 5 drops of sodium thiosulfate solution, recap the bottle firmly and invert 5 times. Place a chlorine detector strip on a dry opened paper towel.

10. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Thoroughly moisten the chlorine detector strip with the aliquot from the glass pipette and immediately flick the chlorine detector strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference chlorine range. A determination must be made within 30 seconds.

IV. PROCEDURE (continued):

11. If no chlorine is detected, recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in the field notebook and place the sample bottle in the ice chest to cool to 4°C. Remember to collect duplicate samples.

12. Continue the process of adding sodium thiosulfate to the sample, recapping, mixing, and testing until no chlorine is detected. Remember to note the number of drops of sodium thiosulfate added to the water sample in the field notebook.

V. SAMPLE TRANSPORT:

After obtaining the water samples, attach the completed chain of custody seal around the plastic cap of each sampling bottle. The samples must be iced or refrigerated at 4°C from the time of collection until extraction and analysis. Endothall is not known to be light sensitive, but excessive exposure to light and heat should be avoided. Always use chopped, grated, or dry ice when chilling the voa samples for transportation. Never use "blue ice" as the samples may not chill adequately. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C upon arrival at the laboratory.

VI. SAMPLE PRESERVATION:

Sample holding studies spanning 14 days indicated that endothall is chemically stable for at least 7 days when preserved with sodium thiosulfate and chilled to 4°C. Samples with unusually high biological activity (e.g. surface water) should be acidified to a pH of ≤ 2 using 1:1 HCl to retard biological degradation of endothall until extraction. Sample extracts should be stored in the dark at 4°C or less. A maximum holding time of 14 days is recommended.

VII. DEFINITIONS:

A. *Sodium Thiosulfate ($Na_2S_2O_3$):* A preservative use to dechlorinate water samples. Reduces free chlorine into acid.

B. *Eluant:* The solvent that contains the analytes after extraction or desorption.

VIII. SAFETY:

The use of protective eyewear and laboratory quality latex gloves is highly recommended when collecting and preserving samples.

IX. SUMMARY OF METHOD:

METHOD 548.1: Liquid-solid extraction (LSE) cartridges containing an intermediate strength, primary tertiary amine anion exchanger are mounted on a vacuum manifold and conditioned with appropriate solvents. LSE disks may be used instead of cartridges providing all quality control criteria specified by the method are met. A 100-ml sample is extracted and the analyte is eluted with 8 ml of acidic methanol. After the addition of a small volume of methylene chloride used as a co-solvent, the dimethyl ester of endothall is formed after approximately 30 minutes with modest heating (50°C). After the addition of salted reagent water, the ester is partitioned into 8-10 ml of methylene chloride. The extraction volume is reduced to 1 ml using a nitrogen purge to achieve a concentration factor of 100. The extract is analyzed by GC/MS or GC/FID with megabore capillary columns.